Influence of Nb Content on Hot Cracking Sensitivity of Weld Heat Affected Zone in Incoloy 800

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Synopsis:

The influence of Nb on the cracking sensitivity of the heat affected zone(HAZ) in Incoloy 800 was examined by hot ductility test using the weld thermal cycle simulater and Spot Varestaint test. The results showed that in each test the cracking sensitivity of Incoloy 800 was increased rapidly when the addition of Nb exceeded about 0.3%, and even in a grater degree when the addition of Nb exceeded about 1%. The cracking can be classified as the liquation cracking caused by constitutional liquation of the second phase particles,mainly NbC and others, in the grain boundary. It was shown,however,that the HAZ cracking sensitivity can be lowered even in Cr-Ni steel containing high Nb, provided that the diameter of the second phase particles such as NbC are made smaller(to less than about 1.8μm).

1. Introduction

In the materials with fully austenitic structure,such as Cr-Ni stainless steel(referred to as Cr-Ni steel) and heat resitant steel,Nb is often added to improve properties at high temperature and resistance to intergranular corrosion. The addition of Nb,however,has a disadvantage that it increases the hot cracking tendency in the heat affected zone(HAZ) in welding.

Since hot cracking in HAZ(HAZ cracking) was pointed out to occur in Type 347 stainless steel[1],a few researchers have taken up this subject[2)~4)],but due to the minuteness of the appearance of the cracks, and to the relative difficulty in detection and to the lack of appropriate test methods,detailed reports on this subject have been very few compared with those on weld metal cracking. On the other hand, Nb's superior contribution to corrosion resistance and high temperature properties in Cr-Ni steels and alloys make this element very atractive,and there are also some types of steel and alloy to which addition of Nb is required. Therefore,it seems quite significant to clarify the HAZ cracking phenomenon in the Nb-containing Cr-Ni steel.

In consideration of the above,Incoloy 800(21%Cr-32%Ni) was chosen for the current study,because it has a fully austenitic structure. Using the thermal cycle simulator,hot ductility tests were conducted,and Spot Varestaint test was also adopted to ascertain the HAZ cracking tendency. Thus,the relationship between the amount of Nb to be added and the HAZ cracking sensitivity was clarified,and the causes of HAZ cracking were discussed.

2. Materials

Table 1 shows the chemical composition of the materials. The materials were cast into 10kg square ingots, and then rolled into bars of 10mm in diameter (for hot ductility test:Samples 1 to 9) or rolled into sheets which are 3.0mm thick(for Spot Varestraint test:Samples 10 to 15),and then they were solution treated at 1100℃. Out of these samples, content of Si in Sample 9 was incresed to 1% in order to examine the influence of Si as well.

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3. Experimental Procedure

3.1 Hot ductility test

The test procedure using the weld thermal cycle simulater was as follows[5]: First, test pieces of 8 mm in diameter were machined from the materials of 10 mm in diameter. Then these were subjected to rapid tensile tests in the thermal cycle on heating and cooling.

Table I Chemical Composition of Incoloy 800 containing Nb, melted., (wt%)

steel No	aim of Nb	C	Si	Mn	P	S	Cr	Ni	Nb	remarks*1
1	—	0.058	0.43	0.76	0.011	0.005	20.89	32.04	—	HD
2	0.05	0.045	0.45	0.72	0.010	0.005	20.93	32.27	0.06	..
3	0.10	0.048	0.46	0.78	0.011	0.008	20.87	31.84	0.12	..
4	0.30	0.050	0.48	0.74	0.012	0.008	20.91	32.48	0.33	..
5	0.50	0.049	0.46	0.76	0.012	0.008	20.91	32.34	0.58	..
6	1.00	0.050	0.46	0.72	0.014	0.008	20.64	32.62	1.09	..
7	2.00	0.052	0.56	0.77	0.011	0.009	20.57	32.04	2.18	..
8	3.00	0.055	0.40	0.66	0.009	0.013	20.61	31.90	2.87	..
9*2	0.30	0.045	1.03	0.74	0.011	0.005	20.89	32.12	0.28	..
10	—	0.033	0.34	0.67	0.005	0.009	20.74	31.65	—	SV
11	0.10	0.032	0.36	0.72	0.005	0.013	20.77	31.95	0.15	..
12	0.30	0.033	0.37	0.73	0.005	0.010	20.70	32.03	0.31	..
13	0.50	0.034	0.38	0.76	0.004	0.013	20.78	31.91	0.60	..
14	1.00	0.034	0.34	0.68	0.004	0.009	20.71	31.96	1.16	..
15	2.50	0.033	0.36	0.72	0.003	0.010	20.59	32.18	2.35	..

*1 HD; for Hot Ductility test, SV; for Spot Varestraint test. *2 Si content aimed 1.0%.

The thermal cycle's heating rate was set at 180℃/sec, and its cooling rate at 80 ℃/sec. The test pieces were kept at the maximum temperature T_{max} for 3 seconds. During heating, the liquation initiation temperature (LIT), which is estimated to take place at about half value of maximum ductility, the nil strength temperature (NST) were obtained, and during cooling, the brittle temperature range (BTR) were obtained. BTR is the temperature range between the solidus and the temperature at which the ductility shows sudden recovery, which is the lower limit of BTR (L.BTR). Then the cracking sensitivity was assessed through the broadness of BTR.

3.2 Spot Varestraint test

The Spot Varestraint tst used in this experiment is a modification of the TIG-A-MAJIG test performed by Goodwin[6]. The plate-shaped test pieces were placed on the bending jig with a certain curvature. Then an arc was generated by TIG welding in the center of the test pieces, and molten pools were created. When the heat input from the arc and the diffusion of heat from molten pool reached an equilibrium in the base metal, force was applied to both ends of the test pieces to bend them along the jig, and the arc was cut off at the same time. The radius of curvature R can be easily changed by choosing different jigs. When the ductility in the HAZ (δ) yields to the strain (ε), or when $\delta < \varepsilon$, cracking is generated.

The strain (ε) added when the test pieces were bent along the bending jig can be represented in the following equation,

$$\varepsilon \fallingdotseq \frac{t}{2R} \times 100\% \quad \text{-------------------------------- (1)}$$

in which "t" stand for thickness of plate (mm) and R for radius of curvature of the bending jig (mm).

The test poeces in this experiment were 60 mm X 140 mm, five each of which was cut out from the 3 mm thick materials. The jig (R=37.5 mm) was chosen to generate a strain of 4% in the test pieces.

After the test, the surface of HAZ was lightly polished and the maximum length of the cracking was measured using a microscope. Also, the area of the brittle HAZ and the total length of the cracking were measured. Each test was conducted five times and the average was adopted as the total length of the cracking.

4. Results

4-1 Rsults of hot ductility test

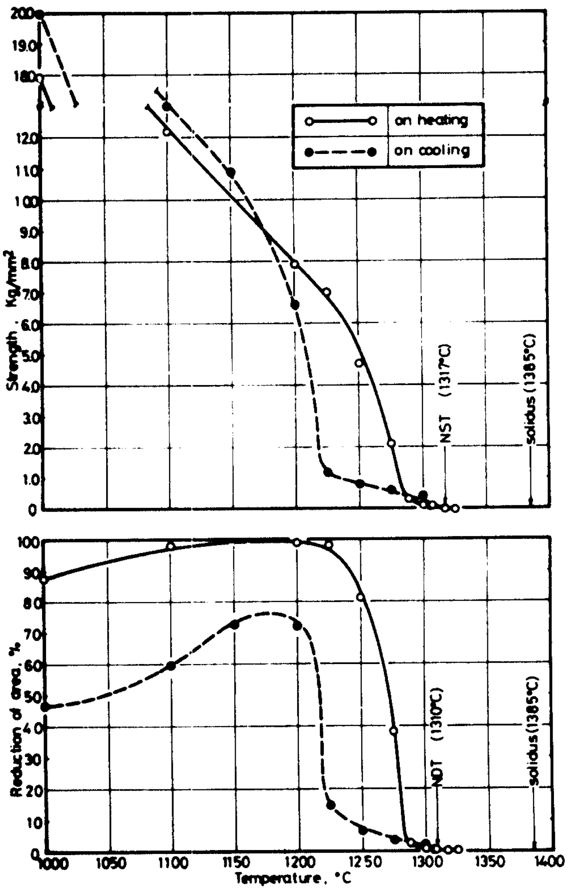
Fig.1 shows an example of test results that used the thermal cycle simulator, and Fig.2 shows the relation between the amount Nb added, LIT, NDT, NST and the solidus.

The tendency of the LIT drop described above suggests that the addition of Nb increases the hazard of liquation cracking.

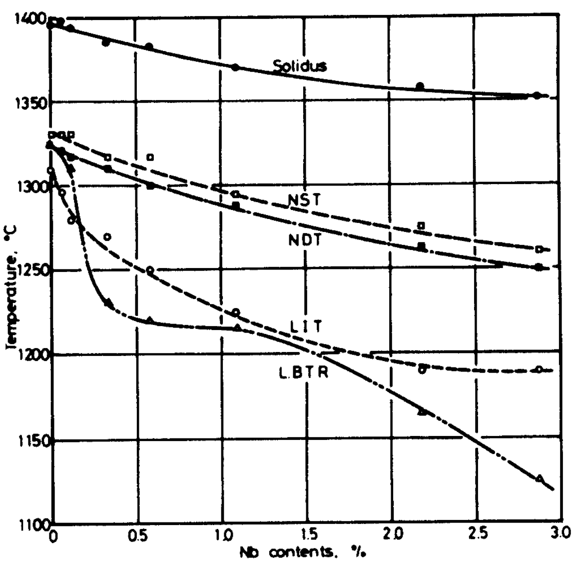
Fig.3 shows the relation between the amount of added Nb and the brittle temperature range(BTR). As the Figure indicate, the BTR of Incoloy 800 when the addition of Nb is less than approximately 0.3% is less than 100℃[7], a range unlikely to cause HAZ cracking by liquation. At 0.3%, the BTR suddenly increases to 160 ℃, indicatig the tendency of the liquation cracking. This tendency is further intensified when the addition of Nb is more than 1%. The sensitivity to the HAZ cracking by liquation measured in term of the BTR is double when 0.3% of Nb is added, and tripled when about 3% of Nb is added.

4-2 Spot Varestraint test results

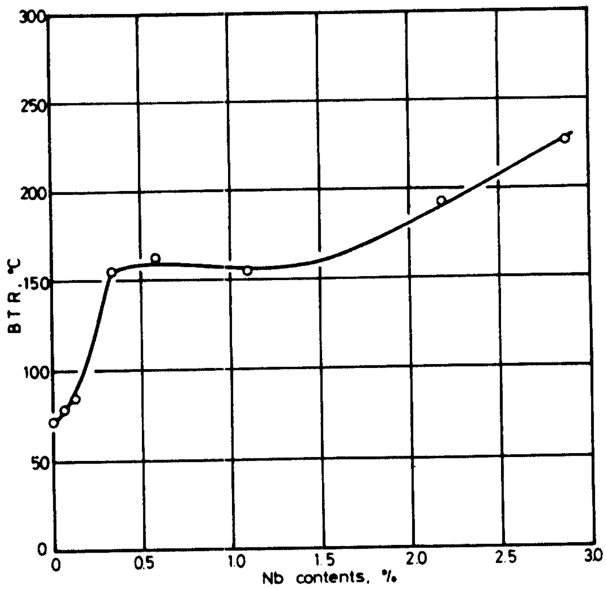
The results of Spot Varestaint test are shown in Fig.4. Fig.4 shows the relationship between Nb content and the total crack length. The results mach well with the results of hot ductility test measured by BTR.



-Figure 1-
Hot ductility characteristics of Incoloy 800 containing 0.33%Nb.



-Figure 2-
Relation between solidus, NST, NDT, LIT, and L.BTR and Nb contents of Incoloy 800.



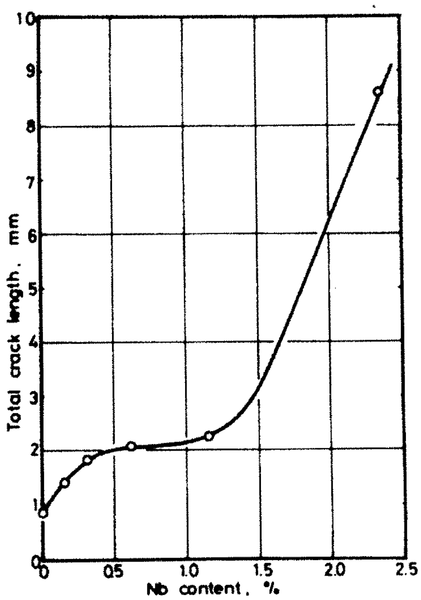
-Figure 3-
Effect of Nb content on BTR of Incoloy 800.

5. Discussion

As was described in Section 4, the influence of the addition of Nb on the cracking sensitivity in Incoloy 800 was examined by the hot ductility test and by Spot Varestaint test, and the results were found to point to nearly identical results, that is, the addition of Nb by about 0.3% rapidly increased the sensitivity, and the addition exceeding 1% further increased the sensitivity.

The fact that the cracking sensitivity increases when addition of Nb is in the vicinity of 0.3% has been pointed out for Type 316 and 310 steel by Gooch and others[8]. It seems that the addition of Nb to high Cr-Ni steel including Incoloy 800 should be kept below 0.3% in consideration of Nb's influence on the cracking sensitivity. Judging from the state of the cracks which seem to be mainly caused by the liquation, the influence of Nb on the cracking sensitivity is suspected to be closely connected with the charactristics of the liquid film in the grain boundary.

For this reason, the characteristics of the liquated grain boundary are discussed somewhat in deail in the following.



-Figure 4-
Relation between total crack length on HAZ and Nb content of Incoloy 800 through spot Varestraint test under 4% augmented strain.

5-1 Microstructure of the liquated grain boundry

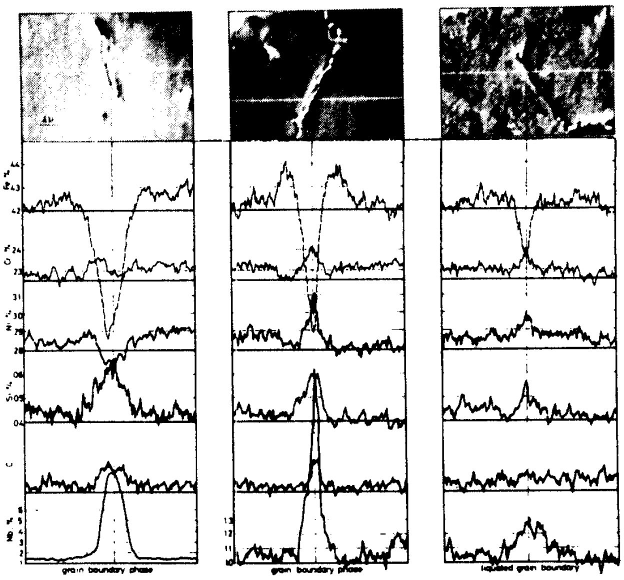
The microstructure of the materials that were heated to NST, shows signs of the grain boundary liquation, but its appearance under the microscope is different from one another depending on the amount of added Nb.

In the material with the least Nb content (0.06%), the widening of the grain boundary is observed which is charactristic of the liquated boundary. In some parts of this boundary, a precipitated phase is found, while other parts show only the trace of liquation. In the latter, the so-called ghost boundary[9] as is often found apart from the actual boundary as a result of the migration of boundary that takes place during the thermal cycle. In the boundary with precipitated phase, on the other hand, the sign of grain boundary migration is not found, suggesting that the precipitated phase serves as an anchor to the migration. The precipitated phase in the liquated boundry is large and eutectic, and depending on the content of Nb, the composition of the phase vary slightly from one another. Thus, it was found out that the liquated grain boundary has two categories, one with precipitated phase and another with only the trace of liquation. The shape and color of the precipitated phase varies considerably depending on the content of Nb and Si. In order to find out the constitution of the liquid film, the line analysis of the liquated boundary described above was conducted using the EPMA.

5-2 Line analysis of liquated boundary by EPMA

The line analysis of the liquated boundary by EPMA was conducted separately for the boundary with only traces of liquation and for the one with precipitated phase. For the material with the highest Nb content, only the boundary with precipitated phase was found, and consequently the analysis was conducted only for this boundary.

An example of the results is shown in Fig.5. The results show that there is considerable difference in the composition of the precipitated phases depending on the content of Nb.



-Figure 5-
Example of scanning electron probe microanalysis of grain boundary phase and liquated grain boundary of Incoloy 800 containing 1.09%Nb, heated to NST.

(1) Liquated boundary

In the liquated boundary without precipitated phase, the enrichment of Cr, Si and Nb was found, but the degree of enrichment did not vary greatly even when the content of Nb was increased. Though it is easy to suppose that the enrichment of the solute element will somewhat lower the melting point in the boundary, there are no data as yet that indicate the extent of such effects. According to the solidus of the Fe-Ni-Cr system reported by Bain and Aborn[10], a 2% increase of Cr in Incoloy 800 composition lowers the solidus by about 10 ℃. In the boundary that contains Si and Nb, etc., the solidus seems to become even lower. In fact, in the material containing 0.06% of Nb with very small amount of the precipitated phase, the lower end of the BTR is about 1320 ℃, or about 80 ℃ lower than the solidus which is 1398℃. This can be attributed to the influence of the enrichment of Cr, Si, Nb, etc. in the grain boundary. All materials showed an enrichment of Si in the liquation boundary, suggesting the relationship between the increase of cracking sensitivity and Si's priority to enrich in the grain boundary.

(2) Precipitated Phase in liquation boundary

The composition of the precipitated phase in the liquation boundary clearly shows an increase of Nb, Si and Ni and a decrese of Cr with the increase of the Nb content. This means that the precipitated phase changes from Cr-dominated compound to ones dominated by Nb, Si and Ni as the content of Nb in the material increases. Especially, in the material containing 0.03% of Nb or more which showed a sudden increase of cracking sensitivity, two kind of precipitations with different compositions were found, suggesting that the precipitated phase has a fairly complex structure.

Table 2 Identification results by extraction replica.

steel no.	Nb contents.(%)	identification results*
2	0.06	Cr_7C_3
4	0 33	NbC**) Cr_7C_3
6	1 09	NbC**) Cr_7C_3 (Ni_3Nb)
8	2 87	NbC**) Ni_3Nb Nb_5Si_3 Cr_7C_3
9	0 28(1 03Si)	NbC**) Cr_7C_3 (Nb_5Si_3)

*) contain inaccuracy

**) They may be Niobium carbonitride, Nb(C.N), because their d_{hkl} coincide with NbN data.too.

As the step following the EPMA analysis described above, the extracted replicas of these materials were made to identify the composition of the precipitated phase by electron diffraction. The results are shown in Table 2. Taking account of both EPMA and electron diffraction, it seems the precipitated phase is dominated by carbide of Cr_7C_3 when the Nb content is as low as 0.06, and NbC starts to dominate when Nb content increases to between 0.33% and 1.09%. When the content of Nb is as high as 2.87%, the precipitated phase is dominated by NbC, Ni_3Nb, Nb_5Si_3, etc. As is noted in section 5-1, every precipitated phase of the liquation boundary is eutectic. The compounds described above are all known to be eutectic products, and the results described above seems to conform to the relationship between the Nb content and the cracking sensitivity mentioned before. The sudden increase of cracking sensitivity when the content of Nb was 0.33% can be attributed to the eutectic of NbC whose eutectic point is 1180 ℃, lower than that of Cr_7C_3 (about 1300 ℃). Further increase of the cracking sensitivity in 2.87%Nb material is attributable to the enrichment of the liquid film that allowed formation of such eutectic product as Ni_3Nb, Nb_5Si_3 as well as NbC, which bring down the eutectic point of the boundary. The increase of the cracking sensitivity in the high Si materials can be explained by the same enrichment in the liquid film resulting in eutectic composition that can form Nb_5Si_3, although this has not been confirmed yet.

Thus, the liquid film has two kinds; one that has solute elements enriched enough to form a precipitated phase, and one that is only slightly more enriched than the matrix and forms an ordinary solid solution. These two kinds can coexist in the same grain boundary as described in 5-1 and 5-2, and the manner of segregation of elements is varied even in the same boundary. The liquation boundary enriched enough to form a precipitated phase is considered to determine the cracking sensitivity. Therefore, if the liquid film is not concentrated enough to form a precipitated phase, or when the enrichment is slight, the HAZ cracking sensitivity in one material seems to be considerably reduced.

5-3 Mechanism of boundary enrichment of solute element and cracking sesitivity

As compared with the microstructures of the specimens heated to NST and as-received ,the former is characterized by the remarkable growth of the grains(especially in the low-Nb specimens), and the second phase dissolved into the matrix.

As a mechanism that explains how the solute element enriches in the HAZ grain boundary and how the boundary's melting point is lowered, the Sweep-up theory[11] that attributes the phenomenon to the migration of boundary is well known. Such boundary can be observed as the ghost boundary[12]. The ghost boundary in the Nb-containing Incoloy 800 was found in low Nb materials, in which the enrichment of Cr, Si, Nb, etc. was clearly observed. But the enrichment of these elements in the ghost boundary did not increase significantly despite the incrase of Nb content, making it rather difficult to think the sweep-up of the solute element by the boundary is the mechanism governing the cracking sensitivity of the Nb-bearing Incoloy 800.

On the other hand, as is described 5-1, no migration of the boundary is observed, in the liquation boundary that has formed a precipitated phase, and the boundary seems to be fixed by the precipitated phasae. Also a comparison with the microstructure of the materials heated to NST and as-received, reveals that the former which underwent the thermal cycle exhibit growth of grains, and also the Nb carbide is solved into the matrix. The appearance of the dissolved Nb carbide reveals that the particles of the Nb carbide are not completely dissolved, but the circumference of the particles forms a contour of diffused area slightly larger than the diameter of the particles. And the center of the particle to have been melted once but remains to form the nucleus of the eutectic products. Such conditions are recognized both in the boundary and in the surrounding area. It looks as if these parts were captured by a migrating boundary, and the precipitated phase looks as if the particles of the carbide melted and penetrated into the grain boundary.

Owczarski[13] and Sullivan[14] closely examined the solution of materials that has a second phase using high Ni-alloy. They reported the results that on rapid heating, the second phase melts at lower temperature than the solidus of the matrix and penetrates into the growing grain boundary, obstructing the growth of the grain and forming in this boundary an enriched segregation of elements that composes the second phaae. It was also reported that the larger the number of such second phase particles,the more they obstruct the growth of the grains and the more they increase the tendency of boundary segregation.

This phenomenon of the second phase particle dissolution reported by Owczarski and Sullivan was next taken up by Savage and Pepe[15] and was theorized into a mechanism of the second phase particle decomposition under non-equilibrium. This mechanism is termed "constitutional liquation". According to this theory,when the material containing the second phase particles is heated faster than the particles'speed of dissolution into the matrix, the boundary between the second phase and the matrix is liquated and finally forms a liquid phase pool. If the material is then rapidly cooled without sufficient diffusion,this liquid pool solidifies at the eutectic point and forms a precipitated phase of the eutectic product.

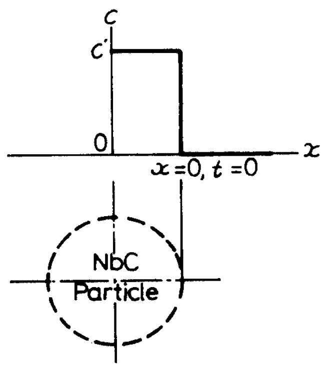
This constitutional liquation mechanism seems to explain successfully the observed state of the carbide particles in Incoloy 800 containing Nb and also the process of generation of the precipitated phase in the boundary. The larger the number of captured second phase particles in the boundary,the larger the boundary area which forms the precipitated phase, which then makes this boundary brittle by wrapping the grains in liquid film when the boundary turns into the liquid phase.

From what is described above, it can be concluded that the cracking sensitivity of Incoloy 800 containing Nb depends on the presence of the second phase particles consisting of mainly NbC, and that the sensitivity increases when enough Nb is added to form the second phase of NbC or others. When,however,the amount of the second phase particles is so small that they decompose and diffuse before constitutional liquation is formed,the cracking sensitivity may possibly drop to the level of the materials that shows only the liquation boundary.

5-4 Constitutional liquation and size of second phase particles

The constitutional liquation is caused by a combination of various factors such as the heating rate,constitution of the second phase particles, the decomposing mechanism of these particles, the lowest solidus temperature and the dissolution of the secondary phase elements into the matrix in the process of diffusion. In order to judge if the constitutional liquation will occur or not, it is necessary to estimate the size of the second phase particles by obtaining the lowest solidus temperatures. However,no such data are as yet available for the second phase consisting of NbC . Therefore it was planned to make a primary approximation of the second phase particles'size enough to cause constitutional liquation,by calculating the distance of diffusion in the cycle.

The following hypotheses were used.
(1) The second phase is NbC. (2) The diffusion of NbC particles is controlled by diffusion coefficient of Nb. (3) The diffusion of Nb into the matrix follows Fick's Low. (4) NbC particles are spherical. (5) The diffusion of NbC progresses with the migration of atoms through interface of the matrix and NbC. (6) The concentration gradient of Nb in the interface of NbC and the matrix is perpendicular when x=0 and t=0 as is shown in Fig.6. The concentration of Nb represented by C' has the following value:when $t = 0$ and $x > 0$, $C = 0$; when $t = 0$ and $x < 0$, $C = C'$ (C' stand for Nb concentration in NbC).



-Figure 6-
Initial and boundary condition for the diffusion of Nb in the vicinity of a NbC particle.

As a rsult, the total length of diffusion x_{tot} is as follows:

$$x_{tot} = \Sigma x_i = \Sigma \sqrt{(D_0 \exp(-Q/RT_i) t_i} \quad \text{------} \quad (2)$$

in which ,D_0;diffuson constant(cm^2/sec), Q;activation energy(cal/mol), R;gas constant(cal/mol.°K), t_i;time at the i-th step when the thermal cycle is divided into n for step function approximation(sec), T_i; temperature at the i-th step when the cycle is divided into n for step function approximation(°K). As a result of the calculation, x_{tot} = 0.73 μm was obtained. Therefore,the minimum diameter of the NbC particle in the thermal cycle is $2x_{tot}$ = 1.46 μm. Actual measurement of the NbC particles of the as-received materials are about 1μm for the material which contains only o.06%of Nb, while those containing 0.33% or more of Nb have particles of about 5 μm, suggesting that the size was enough to cause constitutional liquation. Admittedly,the approximation of the Nb particle size was made under many hypotheses and may deviate from actual size of the particles to some extent, but at least it suggests the possibility that the increase of cracking sensitivity may be reduced if the diameter of the particles in the high Nb materials can be made smaller,in view of the fact that the material No.2 with NbC particles of a diameter of 1 μm or so showed no constitutional liquation. Similarly, on the materials solidified by electroslag melting in which initial carbides are made small, the cracking sensitivity is low[16] ,probably because of the small diameter of the particles of carbide.

6. Conclusion

The examination of the influence of Nb on the HAZ cracking sensitivity in Incoloy 800 by hot ductility test and Spot Varestraint test yielded the following results:

(1) The addition of 0.3% of Nb to Incoloy 800 suddenly increases the HAZ cracking sensitivity,and the sensitivity increases even more when 1% or more of Nb is added.

(2) The cracking can be classified as the liquation cracking caused by the constitutional liquation of the second phase particles consisting of NbC,etc.,which are found in the grain boundary.

(3) The diameter of the NbC particles that do not cause constitutional liquation is approximately calculated to be 3.5 μm. If the diameter of NbC particles can be made smaller than 3.5 μm, there is a possibility that the HAZ cracking sensitivity in the high Nb materials can be reduced.

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